MEMORANDUM

TO: Mary Logan, USEPA

February 28, 2008
Project No.: 933-6154

cc: Sheila Abraham, Ohio EPA
Rainer Domalski, ROC

FR: Julie Lehrman / Steve Finn

RE: NEASE SITE, SALEM, OHIO
ANALYTICAL LABORATORIES FOR MIREX TESTING

On behalf of RÜTGERS Organics Corporation (ROC) this memorandum presents a comparison of mirex analyses undertaken on soil samples from the Nease Site by three different laboratories. The soil samples were collected at the former Nease Site (Operable Unit 2, OU-2) and in the floodplain of the Middle Fork of Little Beaver Creek (Operable Unit 3, OU-3). Analytical testing was undertaken by Ohio EPA Division of Environmental Services, Reynoldsburg, Ohio (DES), Severn Trent Laboratories of North Canton, Ohio (STL),[1] and Exygen Research of State College, Pennsylvania (Exygen).[2]

Analytical Program

A total of 27 composite surface soil samples from OU-2 and 10 composite soil samples from OU-3 were collected between September 18, 2006 and October 6, 2006. In addition, four field duplicates, three matrix spike/matrix spike duplicates (MS/MSD) and two field rinsate blank samples were submitted for quality control purposes. The samples collected, the parameters for analysis, and the laboratory sample identifiers are shown in Table 1. All of the samples were analyzed by DES, with selected samples analyzed by either STL or Exygen as shown in Table 1.

DES prepared the samples using soxhlet extraction with methylene chloride (Ohio Method 581.6), followed by Gel Permeation Chromatography (GPC, Ohio Method 581.2) and filtration through Fluorosil (Ohio Method 581.3) prior to analysis by Gas Chromatography/Electron Capture Detector (GC/ECD) following Ohio Method 590.2.

STL prepared the samples using soxhlet extraction with 1:1 acetone:hexane (SW-846 Method 3540) prior to GC/ECD analysis following SW-846 Method 8081A.

[1] Severn Trent Laboratories, Inc. is now doing business as TestAmerica, Inc.
[2] Exygen Laboratories is a Division of MPI Research and is now doing business as MPI Research.

Exygen prepared the samples by Accelerated Solvent Extraction (ASE, SW-846[3] Method 3545) using 1:1 hexane:acetone solvent, followed by Fluorosil extract clean-up (SW-846 Method 3620B). The solvent was then exchanged to methylene chloride prior to analysis by Gas Chromatography/Electron Capture Negative Ion Mass Spectrometry (GC/ECNIMS) following Exygen Standard Operating Procedure (SOP) 6.2. The GC/ECNIMS detection procedure is analyte specific and is more sensitive than the GC/ECD technology used for conventional pesticide analysis, thereby providing lower reporting limits.

Data Validation Findings

Data validation was performed on the results from all three laboratories following the guidance from USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review (October 1999)/Low Concentration Organic Data Review (June 2001)[4]. While the referenced USEPA National Functional Guidelines were used as guidance during the data review and evaluation procedures, the guidelines strictly apply to CLP Statement of Work (SOW) analyses. Since the samples were analyzed following USEPA SW-846 methods and laboratory specific SOPs, the Quality Control (QC) criteria established in the USEPA National Functional Guidelines are not strictly applicable to the analytical methodologies used. Where the USEPA National Functional Guidelines and method specific criteria differ, professional judgment was used to determine specific qualifications.

In general, chemical results were qualified on the basis of outlying precision or accuracy parameters, or on the basis of professional judgment. The following definitions provide brief explanations of qualifiers that were assigned to data during the data evaluation process.

J	The analyte was reported above the method detection limit; however, the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the method detection limit. The associated quality control measurements indicate a possible low bias.

[3] SW-846 USEPA SW-846 Test Methods for Evaluating Solid Waste may be accessed at URL http://www.epa.gov/epaoswer/hazwaste/test/main.htm

[4] USEPA Contract Laboratory Program National Functional Guidelines EPA-540-R-99-008 and EPA-540-R-00-006 accessed at URL http://www.epa.gov/superfund/programs/clp/guidance.htm

The following bulleted items highlight qualifications for specific QC deficiencies. Although these qualifications were applied to some of the samples collected at the site, the qualifications may not have been required or applied to all samples collected.

- Results from all laboratories were qualified **J/UJ** for sample extraction more than 14 days after sample collection.
- Samples analyzed by Ohio EPA DES and STL were qualified **J** for MS/MSD recovery outside QC limits.
- Samples analyzed by Ohio EPA DES were qualified **J** for field and laboratory duplicate precision outside QC limits.
- Samples analyzed by STL were qualified **J** for surrogate recovery above QC limits.
- Samples analyzed by Exygen were qualified **J** for surrogate recovery below QC limits.

Table 2 presents the validated results for the DES analyses, and Table 3 provides comparative data for DES, STL and Exygen.

Discussion

For screening purposes, Golder used a goal of <30% relative percent difference (RPD) in evaluating the results of split samples analyzed by different laboratories.

STL met this goal for five of the eight samples analyzed, and for all cases where the concentration was greater than the Preliminary Remediation Goal (PRG) of 1,000 ug/kg established for OU-2. In two out of three cases where the PRG was exceeded, STL's result was higher than that of DES. The greatest differences between STL and DES data occurred at the lowest concentrations. In one case (sample SS06-DUP02), the RPD between the DES result and the STL result was 100%. This sample was also submitted to Ohio EPA DES as a blind field duplicate and the RPD between the primary and field duplicate sample results reported by DES was 74%. The discrepancy between the STL and Ohio EPA DES results may therefore be attributed to sample non-homogeneity. As illustrated in Figure 1, the relationship between STL and DES data does not suggest systematic differences.

Exygen met the RPD goal for only two of the eight samples analyzed and the differences between Exygen and DES results appear to increase with concentration.

Conclusion

Based on the data validation and data quality assessment, the DES and STL analytical data for samples collected at the Site were determined to be acceptable for their intended use (including estimated data). Generally acceptable levels of accuracy and precision, based on laboratory control samples (LCS), MS/MSD, field duplicate and surrogate recoveries, were achieved for the data. The data completeness (i.e. the ratio of the amount of valid data obtained to the amount expected, including estimated (**J/UJ**) data) was 100%.

STL and DES data is generally in good agreement, and in two out of three cases where the PRG was exceeded, STL reported higher concentrations than DES. Agency approval is therefore requested to use STL/TestAmerica for future mirex analyses.

TABLE 1
SAMPLE SUMMARY
NEASE CHEMICAL FACILITY
SALEM, OHIO

Operable Unit	Field ID	Matrix	Sample Date	Depth Top (ft bgs)	Depth Bottom (ft bgs)	Mirex	Blank	MS/MSD	Duplicate	DES Lab ID	STL Lab ID	Exygen Analysis ID
OU-2	SB06-A06-HC_00-01_P	SO	9/27/2006	0	1	X				90652		L1970-0003
OU-2	SB06-A07-HC_00-01_P	SO	9/27/2006	0	1	X				90653	A7C050155-001	
OU-2	SB06-A08-HC_00-01_P	SO	9/27/2006	0	1	X				90654		
OU-2	SB06-A09-HC_00-01_P	SO	9/27/2006	0	1	X				90679		
OU-2	SB06-A12-HC_00-01_P	SO	9/27/2006	0	1	X				90655		
OU-2	SB06-A13-HC_00-01_P	SO	9/27/2006	0	1	X				90656		
OU-2	SB06-A17-HC_00-01_D	SO	10/3/2006	0	1	X			X	90673	A7C050155-003	
OU-2	SB06-A17-HC_00-01_P	SO	10/3/2006	0	1	X				90672		L1970-0002
OU-2	SB06-A27-HC_00-01_P	SO	10/4/2006	0	1	X				90674		
OU-2	SB06-A10-HC_00-01_P	SO	10/5/2006	0	1	X				90675		
OU-2	SB06-A11-HC_00-01_P	SO	10/5/2006	0	1	X				90676		
OU-2	SB06-A23-HC_00-01_P	SO	10/5/2006	0	1	X				90677		L1970-0001
OU-2	SB06-A24-HC_00-01_P	SO	10/5/2006	0	1	X				90678	A7C050155-004	
OU-2	SB06-A22-HC_00-01_P	SO	10/6/2006	0	1	X		X		90680	A7C050155-005	
OU-2	SB06-A01-HC_00-01_P	SO	9/28/2006	0	1	X		X		90669		
OU-2	SB06-A02-HC_00-01_D	SO	9/28/2006	0	1	X			X	90658		L1970-0004
OU-2	SB06-A02-HC_00-01_P	SO	9/28/2006	0	1	X				90657	A7C050155-002	
OU-2	SB06-A03-HC_00-01-P	SO	9/28/2006	0	1	X				90659		
OU-2	SB06-A04-HC_00-01_P	SO	9/28/2006	0	1	X				90660		
OU-2	SB06-A05-HC_00-01_P	SO	9/28/2006	0	1	X				90661		
OU-2	SB06-A19-HC_00-01_P	SO	9/29/2006	0	1	X				90662		
OU-2	SB06-A20-HC_00-01_P	SO	9/29/2006	0	1	X				90663		
OU-2	SB06-A21-HC_00-01_P	SO	9/29/2006	0	1	X				90664		L1970-0005
OU-2	SB06-A25-HC_00-01_P	SO	9/29/2006	0	1	X				90665		
OU-2	SB06-A26-HC_00-01_P	SO	9/29/2006	0	1	X				90666		
OU-2	SB06-A14-HC_00-01_P	SO	10/2/2006	0	1	X				90667		
OU-2	SB06-A15-HC_00-01_P	SO	10/2/2006	0	1	X				90668		
OU-2	SB06-A18-HC_00-01_P	SO	10/2/2006	0	1	X				90670		
OU-2	SB06-A16-HC_00-01_P	SO	10/3/2006	0	1	X				90671		
OU-2	SB06-A01-HC_00-01_B	WQ	9/28/2006	0	1	X	X					L9710-001
OU-2	SB06-A17-HC_00-01_B	WQ	10/3/2006	0	1	X	X					L9756-0006
OU-3	SS06-37.5-0-0.5	SO	9/18/2006	0	0.5	X				90714		
OU-3	SS06-35.3-A-0-0.5	SO	9/18/2006	0	0.5	X				90715		
OU-3	SS06-35.3-B-0-0.5	SO	9/18/2006	0	0.5	X			Dup1	90716	A7C050155-006	
OU-3	*SS06-DUP01*	SO	*9/18/2006*	0	0.5	X			X	*90724*		*L1970-0008*
OU-3	SS06-35.0-0-0.5	SO	9/18/2006	0	0.5	X		X		90717		L1970-0006
OU-3	SS06-33.3-0-0.5	SO	9/19/2006	0	0.5	X				90718		
OU-3	SS06-33.0-0-0.5	SO	9/19/2006	0	0.5	X				90719		
OU-3	SS06-22.5-0-0.5	SO	9/19/2006	0	0.5	X				90720		
OU-3	SS06-17.5-0-0.5	SO	9/19/2006	0	0.5	X			Dup2	90721		L1970-0007
OU-3	SS06-DUP02	SO	9/19/2006	0	0.5	X			X	90725	A7C050155-008	
OU-3	SS06-12.5-0-0.5	SO	9/20/2006	0	0.5	X				90722	A7C050155-007	
OU-3	SS06-27.8-0-0.5	SO	9/20/2006	0	0.5	X				90723		

TABLE 1
SAMPLE SUMMARY
NEASE CHEMICAL FACILITY
SALEM, OHIO

Definitions:

OU	Operable Unit
ID	Identifier
SO	Soil
ft bgs	feet below ground surface
STL	Severn Trent Laboratories, Inc.
MS/MSD	Matrix Spike/Matrix Spike Duplicate
DES	Ohio EPA Division of Environmental Services

TABLE 2
OHIO EPA DIVISION OF ENVIRONMENTAL SERVICES DATA
NEASE CHEMICAL FACILITY
SALEM, OHIO

sys_loc_code:		MFLBC-12.5			MFLBC-17.5			MFLBC-17.5			MFLBC-22.5			MFLBC-27.8		
start_depth:		0			0			0			0			0		
end_depth:		0.5			0.5			0.5			0.5			0.5		
sample_date:		09/20/2006			09/19/2006			09/19/2006			09/19/2007			09/20/2006		
sys_sample_code:		SS06-12.5-0-0.5_90722			SS06-17.5-0-0.5_90721			SS06-DUP02_90725			SS06-22.5-0-0.5_90720			SS06-27.8-0-0.5_90723		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	5.7	UJ	5.7	21.5	J	5.6	46.6	J	5.8	321	J	56.6	549	J	132
Solids, Percent	%	69.4			71.6			68.6			70.2			60.1		

sys_loc_code:		MFLBC-33.0			MFLBC-33.3			MFLBC-35.0			MFLBC-35.3			MFLBC-35.3		
start_depth:		0			0			0			0			0		
end_depth:		0.5			0.5			0.5			0.5			0.5		
sample_date:		09/19/2006			09/19/2006			09/18/2006			09/18/2006			09/18/2006		
sys_sample_code:		SS06-33.0-0-0.5_90719			SS06-33.3-0-0.5_90718			SS06-35.0-0-0.5_90717			SS06-35.3-A-0-0.5_90715			SS06-35.3-B-0-0.5_90716		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	1380	J	268	843	J	211	1060	J	276	1560	J	260	2700	J	528
Solids, Percent	%	73.9			74.8			71.9			76.8			75.1		

sys_loc_code:		MFLBC-35.3			MFLBC-37.5		
start_depth:		0			0		
end_depth:		0.5			0.5		
sample_date:		09/18/2006			09/18/2006		
sys_sample_code:		SS06-DUP01_90724			SS06-37.5-0-0.5_90714		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	3010	J	529	811	J	119
Solids, Percent	%	75			67.2		

TABLE 2
OHIO EPA DIVISION OF ENVIRONMENTAL SERVICES DATA
NEASE CHEMICAL FACILITY
SALEM, OHIO

sys_loc_code:		SB06-A01			SB06-A02			SB06-A02			SB06-A03			SB06-A04		
start_depth:		0			0			0			0			0		
end_depth:		1			1			1			1			1		
sample_date:		09/28/2006			09/28/2006			09/28/2007			09/28/2006			09/28/2006		
sys_sample_code:		SB06-A01-HC_00-01_P_90669A			SB06-A02-HC_00-01_P_90657			SB06-A02-HC_00-01_D_90658			SB06-A03-HC_00-01-P_90659			SB06-A04-HC_00-01_P_90660		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	2130	J	485	117	J	38.2	138	J	37.8	97400	J	10700	2130	J	1010
Solids, Percent	%	82			83.6			84			73.8			79.1		

sys_loc_code:		SB06-A05			SB06-A06			SB06-A07			SB06-A08			SB06-A09		
start_depth:		0			0			0			0			0		
end_depth:		1			1			1			1			1		
sample_date:		09/28/2006			09/27/2006			09/27/2006			09/27/2006			09/27/2006		
sys_sample_code:		SB06-A05-HC_00-01_P_90661			SB06-A06-HC_00-01_P_90652			SB06-A07-HC_00-01_P_90653			SB06-A08-HC_00-01_P_90654			SB06-A09-HC_00-01_P_90679		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	668	J	196	41900	J	4730	15800	J	4770	35500	J	4790	4900	J	942
Solids, Percent	%	80.7			84.2			83.2			83.3			84.2		

sys_loc_code:		SB06-A10			SB06-A11			SB06-A12			SB06-A13			SB06-A14		
start_depth:		0			0			0			0			0		
end_depth:		1			1			1			1			1		
sample_date:		10/05/2006			10/05/2006			09/27/2006			09/27/2006			10/02/2006		
sys_sample_code:		SB06-A10-HC_00-01_P_90675			SB06-A11-HC_00-01_P_90676			SB06-A12-HC_00-01_P_90655			SB06-A13-HC_00-01_P_90656			SB06-A14-HC_00-01_P_90667		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	2510	J	521	4300	J	1020	42800	J	4790	782	J	201	332	J	50.2
Solids, Percent	%	76.1			78.5			82.7			79.3			79.6		

TABLE 2
OHIO EPA DIVISION OF ENVIRONMENTAL SERVICES DATA
NEASE CHEMICAL FACILITY
SALEM, OHIO

sys_loc_code:		SB06-A15			SB06-A16			SB06-A17			SB06-A17			SB06-A18		
start_depth:		0			0			0			0			0		
end_depth:		1			1			1			1			1		
sample_date:		10/02/2006			10/03/2006			10/03/2006			10/03/2006			10/02/2006		
sys_sample_code:		SB06-A15-HC_00-01_P_90668			SB06-A16-HC_00-01_P_90671			SB06-A17-HC_00-01_P_90672			SB06-A17-HC_00-01_D_90673			SB06-A18-HC_00-01_P_90670		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	1780	J	495	27400	J	4330	1260	J	255	1100	J	202	311	J	49
Solids, Percent	%	80.8			73.7			78.4			78.4			80.8		

sys_loc_code:		SB06-A19			SB06-A20			SB06-A21			SB06-A22			SB06-A23		
start_depth:		0			0			0			0			0		
end_depth:		1			1			1			1			1		
sample_date:		09/29/2006			09/29/2006			09/29/2006			10/06/2006			10/05/2006		
sys_sample_code:		SB06-A19-HC_00-01_P_90662			SB06-A20-HC_00-01_P_90663			SB06-A21-HC_00-01_P_90664			SB06-A22-HC_00-01_P_90680			SB06-A23-HC_00-01_P_90677		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	188	J	40.4	63.5	J	14.8	66	J	15	165	J	38.6	530	J	98.1
Solids, Percent	%	79.2			80.3			79.3			82.7			81.2		

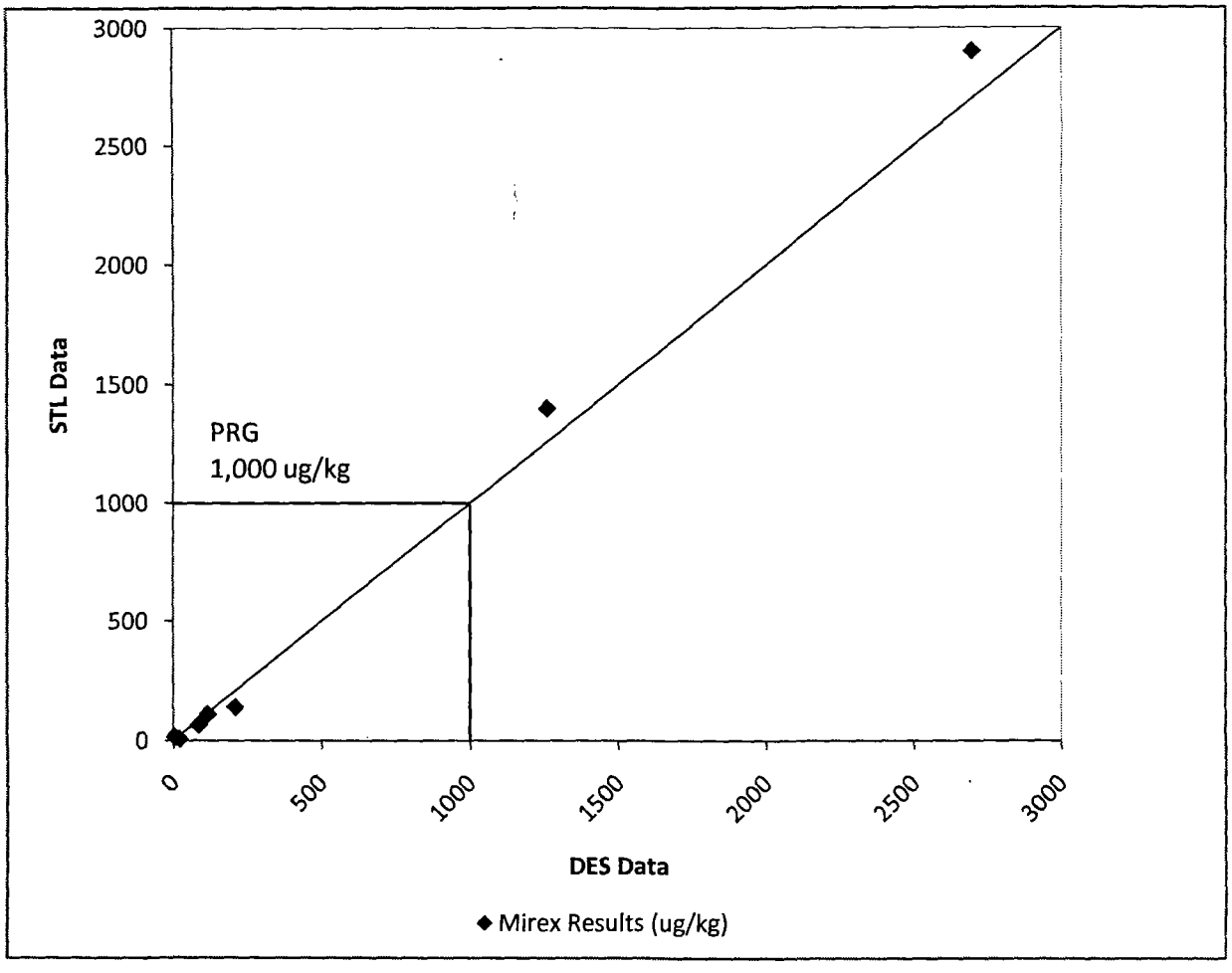
sys_loc_code:		SB06-A24			SB06-A25			SB06-A26			SB06-A27		
start_depth:		0			0			0			0		
end_depth:		1			1			1			1		
sample_date:		10/05/2006			09/29/2006			09/29/2006			10/04/2006		
sys_sample_code:		SB06-A24-HC_00-01_P_90678			SB06-A25-HC_00-01_P_90665			SB06-A26-HC_00-01_P_90666			SB06-A27-HC_00-01_P_90674		
Parameter	**Units**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**	**Result**	**Qual**	**RL**
Mirex	ug/kg	210	J	41.2	134	J	37.9	92.4	J	24.9	913	J	213
Solids, Percent	%	77.2			84.1			79.8			74.8		

TABLE 3
COMPARISON OF SOIL MIREX DATA
NEASE CHEMICAL FACILITY
SALEM OHIO

Golder Sample #	OH DES Sample #	Golder Field Duplicate Sample #	OH DES Field Duplicate Sample #	OH DES Result ug/kg (dry wt)		OH DES Field Duplicate Result ug/kg (dry wt)		Ratio (Primary/Field Duplicate)	RPD (Primary:Field Duplicate)	STL Result ug/kg (dry wt)		Ratio (OH DES/STL)	RPD (OH DES:STL)	Exygen Result ug/kg (corrected to dry wt)		Ratio (OH DES/Exygen)	RPD (OH DES/Exygen)
				Result	**Qual**	**Result**	**Qual**			**Result**	**Qual**			**Result**	**Qual**		
SB06-A06-HC_00-01_P	90652			41900	J									10900	J	3.84	**117.4**
SB06-A07-HC_00-01_P	90653			15800	J					12000	J	1.32	27.3				
SB06-A02-HC_00-01_P	90657	SB06-A02-HC_00-01_D	90658	117	J	138	J	0.85	16.5	110	J	1.06	6.2	93.5	J	1.25	22.3
SB06-A21-HC_00-01_P	90664			66.0	J									23.1	J	2.86	**96.3**
SB06-A17-HC_00-01_P	90672	SB06-A17-HC_00-01_D	90673	1260	J	1100	J	1.15	13.6	1400	J	0.90	10.5	297	J	4.24	**123.7**
SB06-A23-HC_00-01_P	90677			530	J									147	J	3.61	**113.1**
SB06-A24-HC_00-01_P	90678			210	J					140	J	1.50	**40.0**				
SB06-A22-HC_00-01_P	90680			87.4	J					66	J	1.32	27.9				
SS06-35.2-B-0-0.5	90716	SS06-DUP01	90724	2700	J	3010	J	0.90	10.9	2900	J	0.93	7.1	479	J	5.64	**139.7**
SS06-35.0-0-0.5	90717			1060	J									303	J	3.50	**111.1**
SS06-17.5-0-0.5	90721	SS06-DUP02	90725	21.5	J	46.6	J	0.46	**73.7**	7.2	J	2.99	**99.7**	17.3	J	1.24	21.6
SS06-12.5-0-0.5	90722			5.7	UJ					15	J	0.38	**89.9**				

Notes:
Exygen results provided on a wet weight basis. Golder Assoc. calculated the dry weight results using the percent solids measured by OH DES.
RPD = Relative percent difference
OH DES = Ohio Environmental Protection Agency Division of Environmental Services, Reynoldsburg, Ohio
STL = Severn Trent Laboratories, Inc. North Canton, Ohio
Exygen = Exygen Research, State College, Pennsylvania
Samples analyzed by all 3 laboratories are shaded.
RPD > 30% shown in bold font

Qualifier Definitions
UJ = Undetected, estimated reporting limit
J = Estimated


3000
2500
2000
1500
1000
500
0
STL Data
PRG
1,000 ug/kg
0
500
1000
1500
2000
2500
3000
DES Data
◆ Mirex Results (ug/kg)